

Effect of Grain Size on the High Temperature Properties of B2 Aluminides

(NASA-TM-101382) EFFECT OF GRAIN SIZE ON
THE HIGH TEMPERATURE PROPERTIES OF B2
ALUMINIDES (NASA) 26 p CSCL 11F

N89-12720

G3/26 Unclass
0177203

J. Daniel Whittenberger
Lewis Research Center
Cleveland, Ohio

Presented at the
1987 Northeast Regional Meeting "High Temperature Structural
Composites: Synthesis, Characterization and Properties"
cosponsored by The Metallurgical Society of AIME and
The Materials Research Society
Hoboken, New Jersey, May 27-29, 1987



EFFECT OF GRAIN SIZE ON THE HIGH TEMPERATURE PROPERTIES OF B2 ALUMINIDES

J. Daniel Whittenberger
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

ABSTRACT

Measurements of the slow plastic flow behavior of cobalt, iron and nickel B2 crystal structure aluminides have been conducted on materials fabricated by powder metallurgical techniques. Due to this processing, the aluminides invariably had small equiaxed grains, ranging in size from about 3 to 60 μm in diameter. Grain size was dependent on the extrusion temperature utilized for powder consolidation, and it proved to be remarkably stable at elevated temperature. Mechanical properties of all three aluminides were determined via constant velocity compression testing in air between 1000 and 1400 K at strain rates ranging from $\sim 10^{-3}$ to 10^{-7} s^{-1} . At high temperatures and slow deformation rates decreasing the initial grain size was found to lower the flow stress; however the opposite behavior was generally noted for faster straining or lower temperature conditions, where decreasing the grain size increased the strength. FeAl provides the clearest example of grain size strengthening where Hall-Petch behavior appears to be responsible for the effect. In NiAl increases in the flow stresses are only found when the grain size was $\leq 15 \mu\text{m}$; possibly due to a competition between Hall-Petch and subgrain mechanisms. Decreasing the grain size of CoAl appears to be deleterious and just weakens the material. The transition between grain size strengthening and weakening is, most likely, the result of grain boundary sliding and/or diffusional creep which can become important in small grain sized materials under high temperature, slow strain rate conditions.

INTRODUCTION

The nominally equiatomic aluminides of cobalt, iron and nickel possess properties which make them of interest for use both as monolithic and composite matrix materials. In particular CoAl, FeAl and NiAl have the cubic B2 crystal structure, relatively high melting points, exist over a wide range of composition, large solubility for third element additions, and the potential for self protection in oxidizing/corrosive atmospheres. A cubic crystal structure ensures that polycrystalline aggregates can be subjected to cyclic temperature conditions without the danger of cracking due to unequal thermal expansions between adjacent grains. The approximately 1900 K melting point of CoAl and NiAl gives these two aluminides a distinct temperature advantage over current Co-, Fe- and Ni-base alloys. Phase stability, indicated by composition ranges of at least 10 at % Al, and the capacity for substitution of other metallic elements for Co, Fe or Ni, reveal a tolerance for point and possibly line defects and the likelihood of solid solution strengthening. Additionally the "oxidation resistant" coating alloy nature of these aluminides should provide them with the direct ability to survive under the severe conditions found in, for example, gas turbine engines.

In contrast to the considerable research conducted on the elevated temperature behavior of NiAl between 1960 and 1973 (refs. 1 to 9), much less work exists for FeAl (refs. 10 to 14), and only one examination of CoAl (ref. 7) is known. As the majority of these studies involved cast materials with large grain sizes ($>500\text{ }\mu\text{m}$) or single crystals, clear dependencies between microstructure and mechanical strength can not be drawn. However some data exist for NiAl which indicate a possible grain size strengthening effect. In 1960 Grala (ref. 1) noted that (1) the 1088 K tensile strength of Ni-42Al was lessened by a heat treatment which caused grain growth and (2) the addition of small amounts of Mo to Ni-50Al refined the grain size and improved both the 1088 K tensile and stress rupture properties. Likewise in 1965 Lautenschlager, Kiewit and Brittain (ref. 2) reported that the 973 to 1173 K compressive strength of a series of small grain size ($\sim 200\text{ }\mu\text{m}$) NiAl compositions was equal to or greater than that of the large grain size ($\sim 1000\text{ }\mu\text{m}$) forms.

In spite of an apparent grain size - strength relationship in NiAl, no effort has been undertaken to further document, study or exploit this behavior, possibly due to the difficulty of producing small grain size materials via traditional melting and casting techniques. However in 1983 as part of a study of the effect of composition on the elevated temperature slow plastic flow properties of binary aluminides fabricated by powder metallurgy techniques (ref. 15), a correlation between microstructure and flow strength was noted in FeAl (fig. 1) where decreasing the grain size improved the creep resistance at a homologous temperature of ~ 0.7 . Since this initial observation much effort at the Lewis Research Center has been directed at characterizing the elevated temperature strength - microstructure relationships in binary and ternary Co, Fe, and Ni B2 aluminides, and some of the results of this work are outlined in this paper.

EXPERIMENTAL PROCEDURES

Heats of gas atomized -80 or -325 mesh prealloyed powder having the compositions listed in table I were procured from Alloy Metals Inc., Troy, MI. Through mixing appropriate amounts of binary Al-deficient and Al-rich powder in a Vee blender, intermediate CoAl, FeAl and NiAl chemistries were produced. Such blends, as well as the prealloyed binary and ternary intermetallic powders, were fabricated into fully dense materials by hot extruding 51 or 76 mm outside diameter, 6.4 mm thick wall steel cans which had previously been filled with powder and encapsulated under vacuum. Round to round extrusions were undertaken at a 16:1 reduction ratio and temperatures ranging from 1000 to 1505 K; examples of the processing undertaken to densify the aluminide powders in 76 mm cans are given in table II. Because of limitations on extrusion conditions,¹ sections from a few of the extrusions were heat treated to promote grain growth (table II).

¹Lower temperatures were chosen to yield smaller grain sizes while maintaining the extrusion pressure below the press limit of 1310 MPa. The upper limit was dictated by the canning material; above 1505 K the differences in strength between steel and intermetallics would be so great that the aluminide could burst through the can wall.

Cylindrical test specimens 10 mm long with a 5 mm diameter were prepared by electrodischarge machining and grinding, where their length was parallel to the extrusion direction. Samples were taken from both as extruded and heat treated aluminides. Constant velocity compression tests at speeds ranging from 2.12×10^{-3} to 2.12×10^{-6} mm/s were conducted in a universal test machine to ~10 percent or more strain between 1000 and 1400 K in air. The autographically recorded load - time charts were converted to true compressive stresses, strains, and strain rates via the offset method and the assumption of conservation of volume. Additional details concerning specimen fabrication and test procedures can be found in references 15 and 16.

RESULTS

Materials

Both visual and materialographic examination showed that the as extruded and heat treated aluminides were fully dense and crack free; typical microstructures are presented in figure 2. As can be seen in table II, grain size decreases with extrusion temperature, and the microstructures of the lower temperature aluminide extrusions are fairly insensitive to heat treatment. All the materials contained a small amount (<1 percent) of oxides and other second phases probably as a result of powder processing techniques. While x-ray diffraction would occasionally reveal the presence of a slight wire texture ([110] in the 1100 K NiAl extrusions, for example), the majority of the extruded and heat treated aluminides were true polycrystalline materials.

Mechanical Properties

Typical true compressive stress - strain diagrams are presented in figure 3. By far the most common behavior was steady flow where deformation takes place at a more or less constant stress, as illustrated by the data for Fe-41.7Al in figure 3(a). At the slower strain rates, continuous work hardening stress - strain curves (fig. 3(b) for a strain rate $\sim 2.15 \times 10^{-7}$ s⁻¹) were observed in several aluminides, particularly those with a small as extruded grain size. Yielding, generally diffuse occurring over several percent or more of strain, followed by strain softening (Fig. 3(c) for a strain rate $\sim 2.4 \times 10^{-4}$ s⁻¹) was also seen, especially in the cobalt aluminides.

Characteristic true compressive stress - strain rate data for the aluminides are presented in figure 4 where the flow stresses σ and strain rates $\dot{\epsilon}$ were taken from the individual stress - strain diagrams with the following criteria: (1) σ and $\dot{\epsilon}$ are average values from the more or less constant flow regimes; (2) σ and $\dot{\epsilon}$ are taken at about 10 percent strain for continuous work hardening curves; (3) σ and $\dot{\epsilon}$ are means calculated over the diffuse yielding regions.

It is felt that these choices for σ and $\dot{\epsilon}$ are reasonable, and they give valid estimates of the maximum strength properties of the intermetallics. In the case of continuous work hardening, the rate of work hardening had generally diminished to the point that at ~10 percent deformation further straining would only require a slightly higher stress. For completeness in all stress - strain rate diagrams the data from yield points are designated by the half

filled symbols while values from continuous work hardening curves are shown as filled symbols.

All stress - strain rate data were fitted to the standard power law and temperature compensated power law rate expressions

$$\dot{\epsilon} = A\sigma^n \quad (1)$$

and

$$\dot{\epsilon} = B\sigma^n \exp(-Q/RT) \quad (2)$$

where A and B are constants, n is the stress exponent, Q is the activation energy for creep, R is the gas constant and T is the absolute temperature. Fits were accomplished by linear regression techniques, and the results of these calculations for A , B , n , and Q , as well as the standard deviations δ_n and δ_Q for the stress exponent and activation energy respectively, and the coefficients of determination R_d^2 are given in references 16 and 18 for Co-base aluminides, in references 15 and 17 for Fe-base materials and in references 19 and 20 for the B2 Ni-base intermetallics.

INFLUENCE OF GRAIN SIZE ON MECHANICAL PROPERTIES

FeAl

Figure 5(a) presents 1300 K true compressive stress - strain rate data for Fe-39.8Al with different grain sizes. Clearly at the faster strain rates, strength increases as the structure is refined. While the advantage of the $\sim 10 \mu\text{m}$ material lessens, and it even becomes weaker than the $55 \mu\text{m}$ form at the lower strain rates; the intermediate sized material is consistently stronger than the largest grained Fe-39.8Al for all test conditions. The data for the $\sim 10 \mu\text{m}$ aluminide also suggest that two deformation regimes with different n values exist above and below $\dot{\epsilon} \sim 2 \times 10^{-4} \text{ s}^{-1}$. At faster rates the slope of the $\dot{\epsilon} - \sigma$ curve is about the same as that of the larger grain size materials; while at slower rates a much lower stress exponent is evident.

The concept that grain size can affect both strength and deformation mechanism is reinforced by figure 5(b) where the stress - strain rate data for Fe-39.8Al are joined with the results for two other iron aluminides with somewhat similar compositions but differing initial grain sizes. The $43 \mu\text{m}$ diameter Fe-41.7Al behaves as the larger grain sized Fe-39.8Al materials; while the smaller grain sized Fe-43.2Al ($20 \mu\text{m}$) exhibits behavior similar to the $\sim 10 \mu\text{m}$ Fe-39.8Al except the transition point between the two deformation regimes has been shifted to a lower strain rate ($\sim 2 \times 10^{-5} \text{ s}^{-1}$). Furthermore the division of the $\dot{\epsilon} - \sigma$ curves for the smaller grain size aluminides into two regions seems reasonable; as this separation is consistent with the observed changes in the stress - strain diagrams. This is indicated in figure 5(b) where data from continuous work hardening curves (filled symbols) are characterized by $n \sim 3$, and constant flow behavior (open symbols) produces a stress exponent of about 6.

Strengthening. - From figure 5 it is clear that grain size can both strengthen and weaken B2 iron aluminides. When strengthening occurs, it appears that this is simply a manifestation of Hall-Petch behavior with a grain size exponent of 0.5. This is demonstrated in figure 6 where the flow stress

necessary for $\dot{\epsilon} = 2 \times 10^{-5} \text{ s}^{-1}$ is shown as a function of initial grain size d . Assuming that the constant (Friction Stress) in the Hall-Petch description is zero as is suggested by the regression fit of the strength - grain size data in figure 6(b), the dependency between flow stress and grain size implies a strain rate equation of the form

$$\dot{\epsilon} = B' d^p \sigma^n \exp(-Q/RT) \quad (3)$$

where B' is a constant and p is the grain size exponent which should be approximately equal to $n/2$. Fitting the data in figure 5(b) to equation (3) yields

$$\dot{\epsilon} = 1.35 \times 10^{-16} d^{3.09} \sigma^{6.62} \quad (4)$$

where σ is in megapascals and d is in micrometers, and the coefficient of determination is 0.97. The stress - strain rate - grain size data from figure 5(b) can be replotted as stress versus grain size compensated strain rate (fig. 7) to show normal power law behavior and the ability of equation (3) to account for the grain size dependency.

Given that decreasing the grain size can strengthen the B2 iron aluminides under slow strain rate conditions at elevated temperature, examination of the microstructure after testing yielded some unexpected behavior. While little change in average grain dimensions was found in the small grain size material (ref. 15), in several instances the initially 55 μm Fe-39.8Al (fig. 8(a)) underwent extreme grain growth during testing (fig. 8(b)). With such behavior it was, at first, difficult to reconcile the dependency of strength on grain size when the structure is not stable. However examination of other iron aluminides indicated that during grain growth the original high angle grain boundary structure was being replaced by a similar size, low angle grain boundary microstructure (Fig. 9). If high and low angle boundaries are equally efficient dislocation barriers, then in effect a constant "grain size" is maintained during deformation.

Weakening. - Unfortunately, decreasing the grain size does not always strengthen the B2 iron aluminides, and the smaller sized materials eventually become weaker than the large grain structure intermetallics. Data from the regime where grain size weakening occurs are plotted in figure 10; this figure includes results for Fe-46.7Al as well those from figure 5(b) since it had been previously shown that elevated temperature properties of B2 iron aluminides were independent of composition (ref. 17). From figure 10(a) it can be seen that the high temperature strength at low strain rates tends to decrease as the grain size is decreased. Application of equation (3) to these data yields

$$\dot{\epsilon} = 5.83 \times 10^{-8} \sigma^{3.35} d^{-1.01} \quad (5)$$

where the coefficient of determination is 0.99. Replotting the data in figure 10(a) in terms of stress versus grain size compensated creep rate (fig. 10(b)) illustrates the power law behavior, and the capacity of equation (3) to account for the grain size dependency. While this fit on grain size is over, admittedly, a very narrow range, the data suggest that strain rate is inversely proportional to grain size. Thus the grain boundary sliding models presented by Nix (ref. 21) or Langdon and Vastava (ref. 22) which predict $p = -1$ combined with $n = 4$ or $n = 2$ respectively might be applicable.

Although the grain size exponent appears to be close to unity and the stress exponents are greater than one, it is likely that diffusional creep processes are making a contribution to the overall deformation rate. For example the theoretical Nabarro-Herring creep rate is

$$\dot{\epsilon}_{NH} = \frac{12\Omega D\sigma}{kT d^2} \quad (6)$$

where Ω is the atomic volume, D is the lattice diffusion coefficient, and k is Boltzmann's constant. Plots of the Nabarro-Herring diffusional creep rate as a function of stress and grain size are shown in figure 11; comparison of these with the observed behavior (figs. 5 and 10) indicate that under low stress conditions this mechanism can not simply be ignored.

NiAl

Although the two early studies of NiAl (refs. 1 and 2) presented data which showed an apparent grain size effect on mechanical properties, it was recently concluded (ref. 19) that the 1200 to 1400 K creep strength of NiAl was independent of grain structure for grain sizes $\geq 15 \mu\text{m}$. This conclusion was drawn on the basis of the existing mechanical data and the observation of subgrain formation in NiAl during creep. It was predicted, however, that microstructural strengthening could occur if the grain size was less than the equilibrium subgrain diameter. Quite possibly this criteria was met by Vedula, Pathare, Aslandis and Titran (ref. 24) who found a doubling in the strength of Ni-50Al at 1300 K and $\dot{\epsilon} \approx 2 \times 10^{-6} \text{ s}^{-1}$ from ~ 15 to 33 MPa when grain size was decreased from 48 to 2 μm .

Although no quantitative data exists on the size of subgrains formed in NiAl, the recent review and analysis of subgrain diameter - stress relationships by Raj and Pharr (ref. 25) indicates that the average subgrain size d_s can be estimated to within a factor of two from

$$d_s = \frac{23bG}{\sigma} \quad (7)$$

where b is the magnitude of the Burgers vector and G is the shear modulus. Values of the estimated subgrain size are tabulated as a function of stress in table III; for all practical purpose they are independent of temperature since the only temperature dependent term in equation (7) is the shear modulus. As the observed flow stresses in NiAl tested between 1200 and 1400 K and strain rates less than 10^{-4} s^{-1} are on the order of 30 MPa (ref. 19), the data in table III clearly indicate that grain sizes $\leq 20 \mu\text{m}$ would be necessary for grain size strengthening.

A recent study (ref. 20) has examined the effect of grain size on the slow plastic deformation behavior of Ni-48.25Al between 1000 and 1400 K for $d \leq 20 \mu\text{m}$, and stress - strain rate plots illustrating the behavior at 1100 and 1300 K are given in figure 12. Without question refining the microstructure can increase the strength; however the behavior is not consistent:

(1) The strengths of the 9 and 18 μm materials are similar at 1100 K and $\dot{\epsilon} \sim 2 \times 10^{-4} \text{ s}^{-1}$.

(2) The 5 μm aluminide is stronger than the 9 μm form at 1100 K and $\dot{\epsilon} \sim 2 \times 10^{-4} \text{ s}^{-1}$, while at $\dot{\epsilon} \sim 2 \times 10^{-7} \text{ s}^{-1}$ the opposite is true.

(3) At 1300 K the behavior of both small grain size intermetallics is similar and exceeds the strength of the 18 μm form, but there is a significant drop off in strength under $\dot{\epsilon} \sim 2 \times 10^{-7} \text{ s}^{-1}$ test conditions.

From the data in table III the boundaries between possible grain size strengthening and no effect are approximately 120, 70, and 35 MPa respectively for 5, 9, and 18 μm structures. Thus, for example, a 9 μm grain size could strengthen at stresses below 70 MPa (the normal, stable subgrain diameter should be larger than 9 μm , hence less than the grain size) but not above this value. This concept provides a reasonable explanation for the essentially equal strengths of the 9 and 18 μm materials at 1100 K - $\dot{\epsilon} \sim 2 \times 10^{-4} \text{ s}^{-1}$ test conditions while the 5 μm form could be stronger (fig. 12). The behavior of the aluminides at 1300 K and $2 \times 10^{-6} < \dot{\epsilon} < 2 \times 10^{-4} \text{ s}^{-1}$ is also explicable on the basis of the subgrain - grain size argument and the observation that the originally 5 μm material suffered grain growth to approximately 10 μm during testing (fig. 12, ref. 20). Both smaller grained forms have about the same average size during testing, hence nearly equivalent strengths; however they should be stronger than the $\sim 18 \mu\text{m}$ material due to the grain size effect.

While little can be quantitatively predicted concerning grain boundary sliding rates in the power law regime for NiAl, it can be shown that part of the weakening of the 5 μm form at 1100 K - $\dot{\epsilon} \sim 2 \times 10^{-7} \text{ s}^{-1}$ and both nominally 10 μm materials at 1300 K - $\dot{\epsilon} \sim 2 \times 10^{-7} \text{ s}^{-1}$ could be due to diffusional creep processes. Theoretical Nabarro-Herring creeps rates calculated from equation (7) for NiAl at 1300 K are shown in figure 13 as a function of grain size; comparison of these predictions with the actual 1300 K stress - strain rate behavior in figure 12 reveal that diffusional mechanisms can contribute to the overall deformation rate when low strain rates and small microstructures are considered.

CoAl

In general little information exists on the effect of grain structure on the slow plastic deformation behavior of CoAl-based materials at elevated temperatures because powder metallurgy techniques have yielded only small grain sizes, and these have proven to be quite resistant to grain growth (ref. 16). Some data do exist for the B2 ternary aluminide Co-14.6Ni-43.8Al tested between 1100 and 1400 K over a very limited grain size range, and typical stress - strain rate results are presented in figure 14. At 1100 K the microstructure was stable, and as can be seen in figure 14(a), the flow strength basically decreased with decreasing grain size. Similar behavior exists at 1300 K; however grain growth did take place. This resulted in both smaller forms possessing approximately the same size after testing and hence nearly equivalent strengths.

Results from testing of binary alloys, while meager, also tend to support the contention that increasing the grain size increases the elevated temperature strength. It was noted that a Co-48.5Al specimen which underwent grain

growth during testing at 1400 K was much stronger than expected (ref. 16). Additionally the strengths reported by Hocking, Strutt and Dodd (ref. 7) for Co-50.4Al single crystals were much greater than those measured in $\sim 10 \mu\text{m}$ materials (ref. 16).

It is not at all clear why the elevated temperature slow plastic flow properties of CoAl are reduced by decreasing the grain size. While the grain sizes for the CoAl-based materials are somewhat smaller than those for the other aluminides, this difference in itself should not be sufficient to so greatly enhance the roles of diffusional creep and grain boundary sliding as the rates of lattice diffusion in CoAl (ref. 28) are about one order of magnitude lower than those for NiAl.

Extensive subgrain formation has been observed in 1505 K extruded Co-49.3Al by Yaney and Nix (ref. 29), and they found that this substructure was difficult to replace during lower temperature deformation. In essence the Co-49.3Al possessed a subgrain size which reflected the approximately 1310 MPa pressure required for extrusion of these materials; from equation (7) and the elastic modulus data of Harmouche and Wolfenden (ref. 30), this high force would generate an estimated average subgrain size of $\sim 0.5 \mu\text{m}$. If this is the case for all extruded CoAl-based materials, then grain size strengthening, for all practical purposes, could never be observed, for refinement of the grain microstructure less than a half a micron would be required.

DISCUSSION

Study of the effect of microstructure on the slow plastic flow properties of the three B2 crystal structure aluminides CoAl, FeAl, and NiAl at elevated temperature indicates that these intermetallics can deform by several mechanisms. This means that a single strategy to obtain optimum properties for all three materials will not work; rather the microstructure has to be tailored for each intended use. Furthermore, due to a variety of deformation modes operating in each aluminide, each system probably exhibits only a small range of temperature - microstructure conditions where useful mechanical properties can be developed.

FeAl can be strengthened by decreasing the grain size; however this mechanism is counteracted under high temperature - slow strain rate conditions, and the material becomes weaker as the microstructure is refined. Thus it would appear that reducing the grain size might be an effective method to improve the long term mechanical properties at lower temperatures (perhaps 1000 K) where grain boundary sliding and diffusional creep processes are slow.

Because creep in NiAl is related to the formation of subgrains, reducing the grain diameter to sizes smaller than the normally expected subgrain dimensions strengthens the material. However instability of the microstructure and possible contributions via grain boundary sliding and/or diffusional creep mechanisms would seemingly limit this approach at high temperatures. While the concept can and should be applied at lower temperatures, there is probably a lower temperature limit below which little advantage could be gained. Such a limit would evolve from the natural increase in strength of a material with decreasing temperature and the decrease in equilibrium subgrain size as the strength of the intermetallic increases (eq. (7)). One possible superiority of

a small grain size at any temperature, though, would be its immediate effect; whereas the equilibrium subgrain structure and associated strength can only be developed through deformation.

Clearly for values less than 10 μm decreasing the grain size of CoAl-base materials reduces the elevated temperature, slow strain rate strength; thus one would want to avoid the microstructures which seem advantageous to FeAl and NiAl.

Obviously much experimental work needs to be done to define specific deformation modes in each material. Questions which need to be answered include:

CoAl, FeAl, and NiAl

1. What is inhibiting diffusional creep mechanisms in the small grain size forms of all three aluminides?
2. How do mechanical properties measured under tensile creep conditions (cavitation possible) compare to those determined in compression (cavities generally not observed)?

FeAl -- Grain Size Strengthening Regime

1. Is grain size strengthening a result of Hall-Petch behavior, traditionally believed to be a low temperature (fast strain rate) process, or is it a manifestation of Sherby-Klundt-Miller (ref. 31) creep model where the subgrain size is replaced by the grain diameter (ref. 19)?
2. Does the subgrain structure that develops (a) in large grain size materials and (b) during deformation induced grain growth comply with equation (7) or is the average size of such structures dictated by the original grain dimensions?
3. The activation energy for this regime (~ 460 kJ/mol, refs. 15 and 17) is much higher than that reported for diffusion (~ 300 kJ/mol, ref. 23). What does this mean in terms of active deformation mechanisms?

FeAl -- Grain Size Weakening Regime

1. Is the grain size exponent truly -1 , or is it an aberration due to the very limited range of grain sizes tested to date?
2. The activation energy for this regime (~ 400 kJ/mol, refs. 15 and 17) is also much higher than that reported for diffusion (~ 300 kJ/mol, ref. 23). What does this mean in terms of active deformation mechanisms? Does such a high activation energy negate Nix's grain boundary sliding model (ref. 21) which predicts that grain boundary diffusion is controlling rather than lattice diffusion?

NiAl

Do grain boundary sliding processes take place?

CoAl

1. As Co-49.3Al has been shown to be much stronger than the Al deficient CoAl materials (ref. 16), do the same deformation processes occur in these aluminides?

2. Is grain boundary sliding partially responsible for the poor strength of the fine grain size materials?

SUMMARY OF RESULTS

Studies of the elevated temperature slow plastic deformation behavior of the B2 crystal structure intermetallics CoAl, FeAl and NiAl have shown that strength is dependent on the grain structure. While FeAl and NiAl can be strengthened by decreasing the grain size, for CoAl-base aluminides grain refinement below $\sim 10 \mu\text{m}$ weakens the material. Strengthening of NiAl can result when the grain size is less than the usual subgrain size which would be established by the applied stress; unfortunately under elevated temperature - slow strain rate conditions diffusional creep and/or grain boundary sliding mechanisms and grain growth can occur which reduce the effectiveness of the small microstructures. FeAl possesses two distinct deformation regimes where decreasing the grain size can either produce strengthening or weakening. Hall-Petch behavior seems responsible for the strengthening while grain boundary sliding is a likely cause of weakening.

REFERENCES

1. E.M. Grala: in Mechanical Properties of Intermetallic Compounds, J.H. Westbrook, ed., Wiley, New York, 1960, pp. 358-404.
2. E.P. Lautenschlager, D.A. Kiewit, and J.O. Brittain: Trans. Met. Soc. AIME, 1965, vol. 233, pp. 1297-1302.
3. A.G. Rozner and R.J. Wasilewski: J. Inst. Met., 1966, vol. 94, pp. 169-175.
4. A. Ball and R.E. Smallman: Acta Met., 1966, vol. 14, pp. 1349-1355.
5. R.R. Vandervoort, A.K. Mukherjee, and J.E. Dorn: Trans. ASM, 1966, vol. 59, pp. 930-944.
6. R.T. Pascoe and C.W.A. Newby: Met. Sci., 1968, vol. 2, pp. 138-143.
7. L.A. Hocking, P.R. Strutt, and R.A. Dodd: J. Inst. Met., 1971, vol. 99, pp. 98-101.
8. W.J. Yang and R.A. Dodd: Met. Sci., 1973, vol. 7, pp. 41-47.

9. H.L. Fraser, R.E. Smallman, and M.H. Loretto: Philos. Mag., 1973, vol. 28, pp. 651-665.
10. A. Lawley, J.A. Coll, and R.W. Cahn: Trans. Met. Soc. AIME, 1960, vol. 218, pp. 166-177.
11. G. Sainfort, P. Mouturat, P. Pepin, J. Petit, G. Cabane, and M. Salesse: Mem. Sci. Rev. Metall., 1963, vol. 60, pp. 125-134.
12. J.P. Cottu, P. Moine, P. Grosbras, J. Caisso, and R. Jacquesson: Mem. Sci. Rev. Metall., 1966, vol. 63, pp. 793-799.
13. Y. Umakoshi and M. Yamaguchi: Philos. Mag. A, 1980, vol. 41, pp. 573-588.
14. Y. Umakoshi and M. Yamaguchi: Philos. Mag. A, 1981, vol. 44, pp. 711-715.
15. J.D. Whittenberger: Mater. Sci. Eng., 1983, vol 57, pp. 77-85.
16. J.D. Whittenberger: Mater. Sci. Eng., 1985, vol. 73, pp. 87-96.
17. J.D. Whittenberger: Mater. Sci. Eng., 1986, vol. 77, pp. 103-113.
18. J.D. Whittenberger: Mater. Sci. Eng., 1987, vol. 85, pp. 91-99.
19. J.D. Whittenberger: J. Mater. Sci., 1987, vol. 22, pp. 394-402.
20. J.D. Whittenberger: J. Mater. Sci., 1988, vol. 23, pp. 235-240.
21. W.D. Nix: in Rate Processes in Plastic Deformation of Materials, J.E Dorn, J.C.M. Li, and A.K. Mukherjee, eds., American Society for Metals, Metals Park, OH, 1975, pp. 384-409.
22. T.G. Langdon and R.B. Vastava: in Mechanical Testing for Deformation Model Development, ASTM-STP-765, R.W. Rohde and J.C. Swearingen, eds., ASTM, Philadelphia, PA, 1982, pp. 435-451.
23. L.N. Larikov, V.V. Geichenko, and V.M. Fal'chenko: Diffusion Processes in Ordered Alloys, Published for the National Bureau of Standards and National Science Foundation, Washington, DC, by Auerind Publishing Co. Pvt. Ltd., New Delhi, India, 1981.
24. K. Vedula, V. Pathare, I. Aslanidis, and R.H. Titran: in High-Temperature Ordered Intermetallic Alloys, C. Koch, C.T. Liu, and N.S. Stoloff, Materials Research Society, Pittsburgh, PA, 1984, pp. 411-421.
25. S.V. Raj and G.M. Pharr: Mater Sci. Eng., 1986, vol. 81, pp. 217-237.
26. M.R. Harmouche and A. Wolfenden: J. Test. Eval., 1987, vol. 15, pp. 101-104.
27. G.F. Hancock and B.R. McDonnell: Phys. Stat. Sol. A, 1971, vol. 4, pp. 143-150.
28. F.C. Nix and F.E. Jaumot Jr.: Phys. Rev., 1951, vol. 83, pp. 1275-1276.

29. D.L. Yaney and W.D. Nix, personal communication, Stanford University, Stanford, CA, 1988.
30. M.R. Harmouche and A. Wolfenden: Mater. Sci. Eng., 1986, vol. 84, pp. 35-42.
31. O.D. Sherby, R.H. Klundt, and A.K. Miller: Met. Trans. A, 1977, vol. 8, pp. 843-850.

TABLE I. - COMPOSITION OF B2 ALUMINIDES

ID	Amount, at %							
	Al	Co	Fe	Ni	C	H	N	O
Co-44.9Al	44.9	bal	----	----	0.03	0.05	0.03	0.12
Co-51.3Al	51.3	bal	----	----	.028	.055	.004	.12
CoFeAl	44.6	bal	15.4	----	.015	.022	.019	.011
CoNiAl	43.8	bal	----	14.6	.019	.05	.006	.053
Fe-41.7Al	41.7	---	bal	----	.2	.05	.002	.06
Fe-48.7Al	48.7	---	bal	----	.017	.045	.005	.15
FeAl	39.8	---	bal	----	----	----	----	----
Ni-43.9Al	43.9	---	----	bal	.011	.02	.004	.09
Ni-52.7Al	52.7	---	----	bal	.01	.05	.004	.14
NiAl	48.25	---	----	bal	.003	----	----	.07

TABLE II. - THERMOMECHANICAL PROCESSING SCHEDULES AND INITIAL GRAIN SIZES

ID	Extrusion temperature, K	Heat treatment	Equiaxed grain size, μm
CoFeAl-II	1505	-----	10
CoFeAl-IV	1435	-----	5.5
CoNiAl-III	1250	-----	2.5
CoNiAl-IIIt	1250	16 h @ 1300 K	5.5
CoNiAl-IV	1505	-----	8
FeAl-I	1505	-----	55
FeAl-Iht	1505	16 h @ 1535 K	185
FeAl-II	1200	-----	9
FeAl-IIht	1200	16 h @ 1300 K	11
NiAl-I	1505	-----	18
NiAl-II	1100	-----	5
NiAl-IIht	1100	16 h @ 1390 K	9

ORIGINAL PAGE IS
OF POOR QUALITY

TABLE III. - ESTIMATED
SUBGRAIN DIAMETERS
IN NiAl^a

Stress, MPa	d_s , μm
300	2
120	5
60	10
30	20
15	40
10	60
7.5	80
6	100
2	300

^a $b = 2.9 \times 10^{-4} \mu\text{m}$,
 $G \approx E/2.6$ where
Young's modulus
data E from
ref. 26.

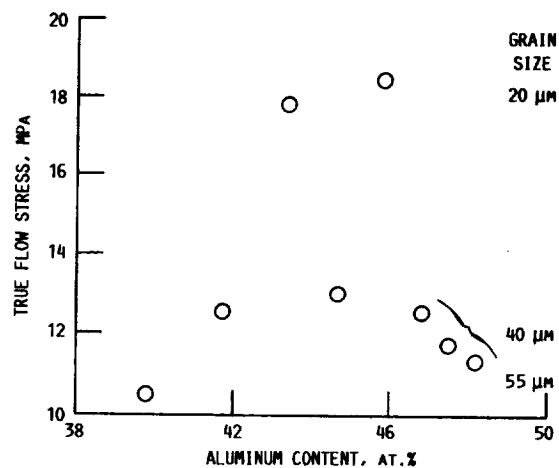


FIGURE 1. - FLOW STRESS TO PRODUCE A STRAIN RATE OF $\sim 2 \times 10^{-4} \text{ s}^{-1}$ AT 1300 K IN POLYCRYSTALLINE B2 CRYSTAL STRUCTURE FeAl AS A FUNCTION OF COMPOSITION WHERE THE GRAIN SIZE OF (1) Fe-39.8Al WAS 55 μm ; Fe-43.5Al AND Fe-45.7Al WERE ABOUT 20 μm ; AND THE REMAINING INTERMETALLICS WERE $\sim 40 \mu\text{m}$, REF. 15.

ORIGINAL PAGE IS
OF POOR QUALITY

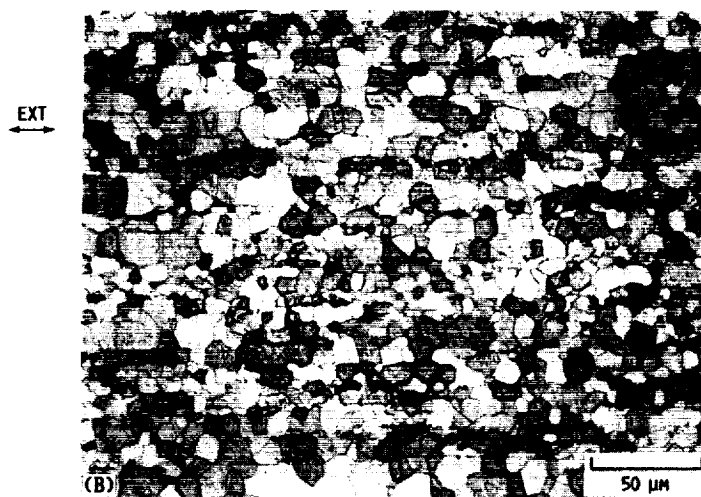
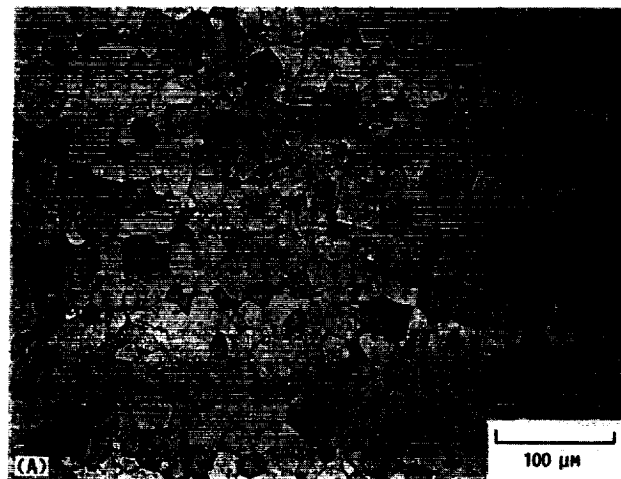
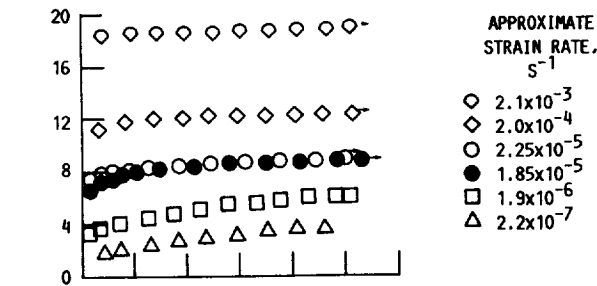
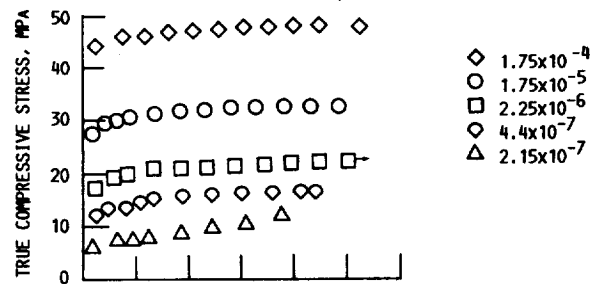


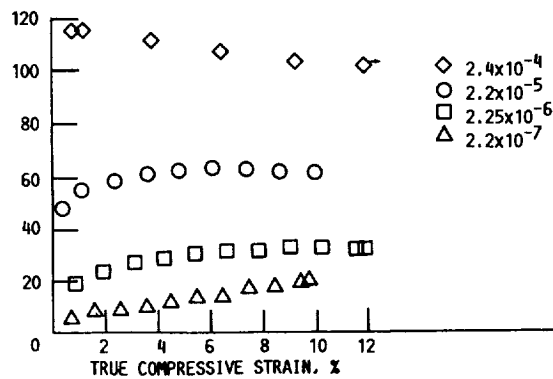
FIGURE 2. - GRAIN STRUCTURES OF (A) AS EXTRUDED Ni-52.7Al AND
(B) HEAT TREATED CoNiAl-III, Ni-52.7Al ETCHED WITH 33 HCl,
HNO₃ AND 33 ACETIC ACID (PARTS BY VOLUME); CoNiAl-III ETCHED
WITH MARBLE'S REAGENT.



(A) Fe-41.7Al EXTRUDED AT 1505 K.
INITIAL GRAIN SIZE OF $43 \mu m$.



(B) Ni-50.6Al EXTRUDED AT 1420 K.
INITIAL GRAIN SIZE OF $16 \mu m$.



(C) CoFeAl EXTRUDED AT 1435 K.
INITIAL GRAIN SIZE OF $5.5 \mu m$.

FIGURE 3. - TYPICAL 1300 K TRUE STRESS-STRAIN DIAGRAMS.

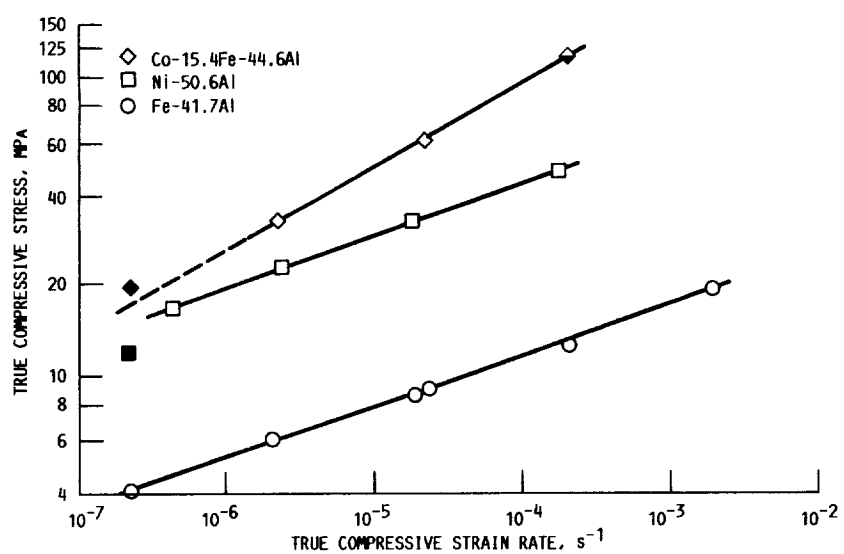


FIGURE 4. - TYPICAL TRUE STRESS - STRAIN RATE CURVES FOR B2 ALUMINIDES. DATA TAKEN FROM STRESS - STRAIN DIAGRAMS SHOWN IN FIG. 3.

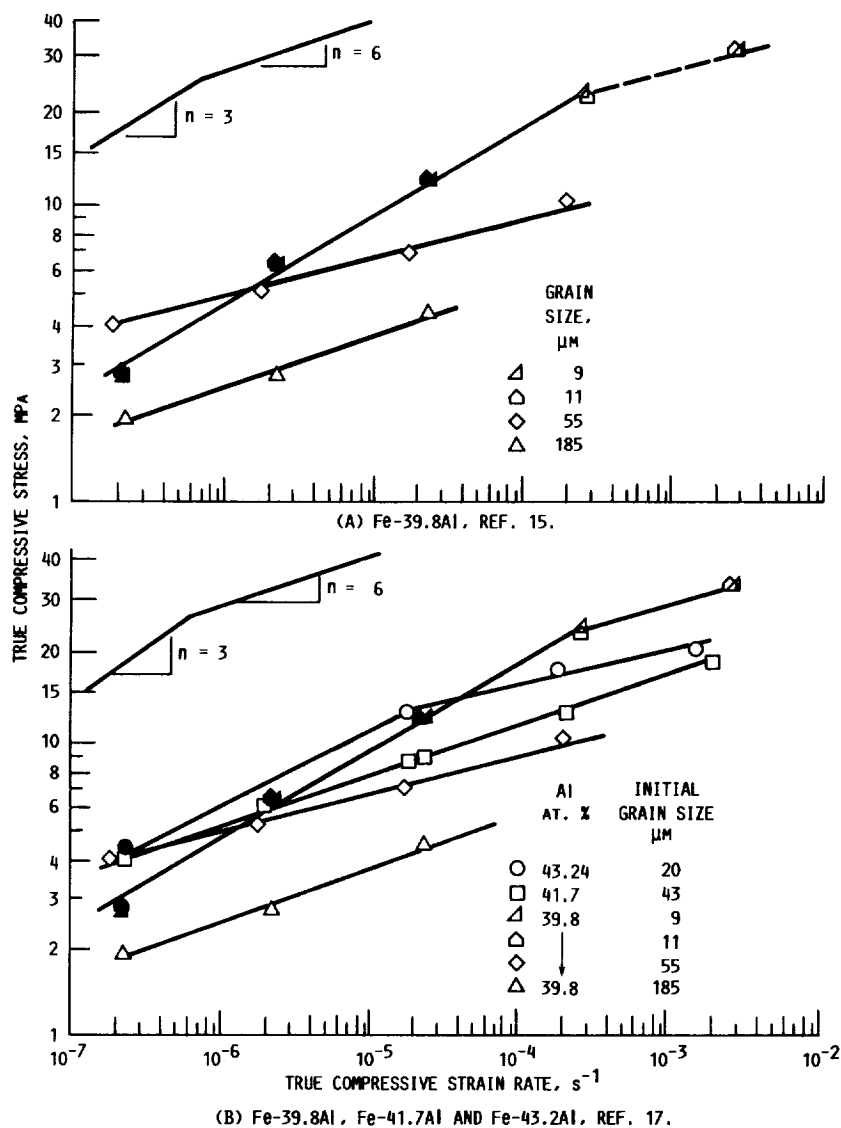


FIGURE 5. - TRUE COMPRESSIVE FLOW STRESS - STRAIN RATE BEHAVIOR AT 1300 K FOR B2 IRON ALUMINIDES SUBJECTED TO SEVERAL THERMOMECHANICAL PROCESSING SCHEDULES WHICH VARIED THE INITIAL GRAIN SIZE.

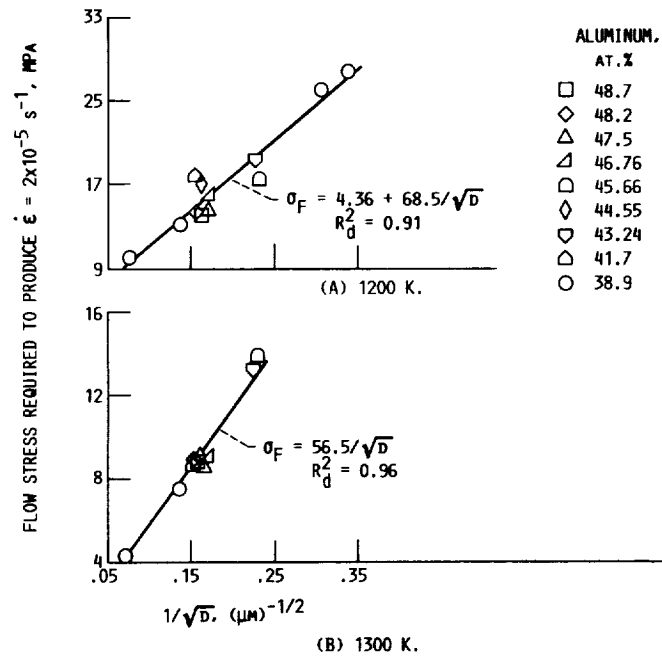


FIGURE 6. - FLOW STRESSES NECESSARY TO PRODUCE A STRAIN RATE OF $2 \times 10^{-5} \text{ s}^{-1}$ AS A FUNCTION OF GRAIN SIZE FOR B2 IRON ALUMINIDE TESTED IN A REGIME WHERE GRAIN REFINEMENT INCREASES STRENGTH, REF. 17.

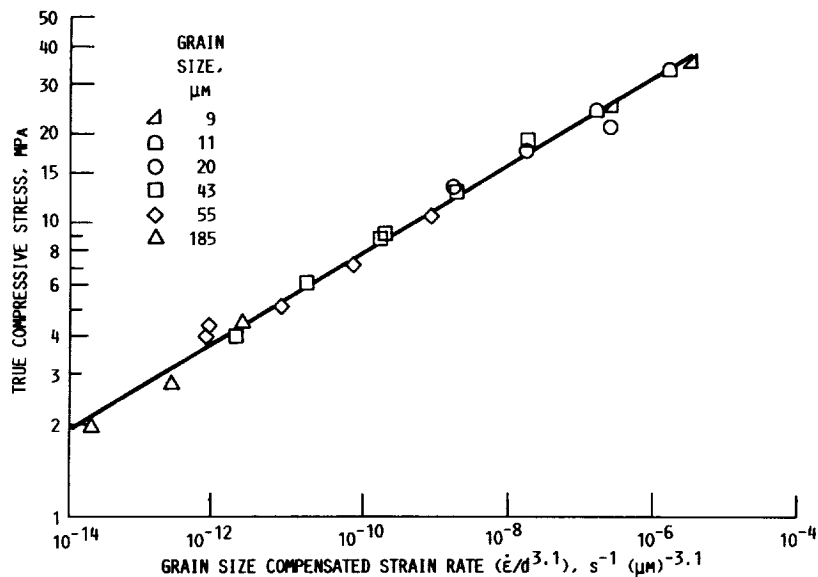
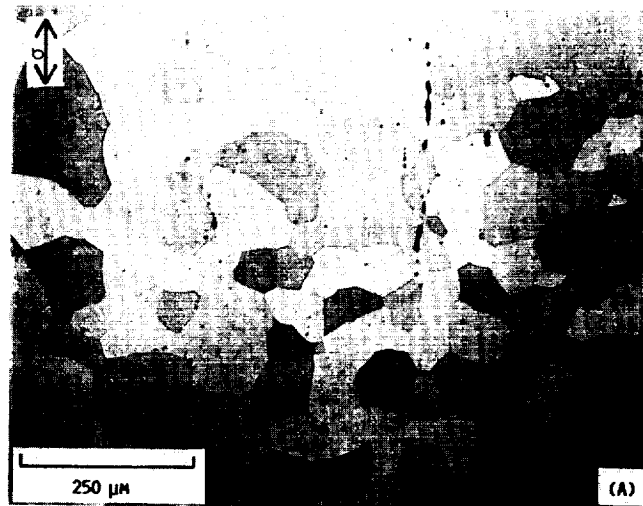
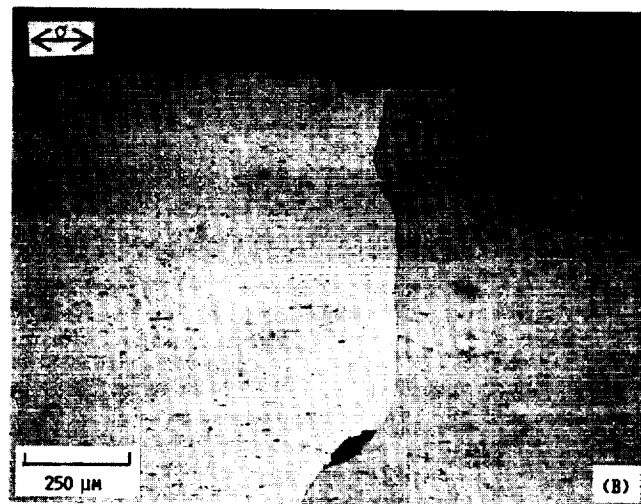


FIGURE 7. - FLOW STRESS AS A FUNCTION OF GRAIN SIZE COMPENSATED STRAIN RATE FOR Fe-39.8Al, Fe-41.7Al AND Fe-43.2Al TESTED AT 1300 K.

ORIGINAL PAGE IS
OF POOR QUALITY



(A) 1200 K AND $\dot{\epsilon} = 1.7 \times 10^{-6} \text{ s}^{-1}$ TO 9.2 PERCENT STRAIN.



(B) 1300 K AND $\dot{\epsilon} = 1.7 \times 10^{-6} \text{ s}^{-1}$ TO 11.3 PERCENT STRAIN.

FIGURE 8. - TYPICAL PHOTOMICROGRAPHS OF INITIALLY 55 μm Fe-39.8Al AFTER COMPRESSION TESTING. MATERIALS SWAB ETCHED WITH A MIXTURE OF 33 ACETIC ACID, 33 HCl, 33 HNO₃ AND 1 HF (PARTS BY VOLUME). REF. 15.

LARGE ANGLE ← | → SMALL ANGLE

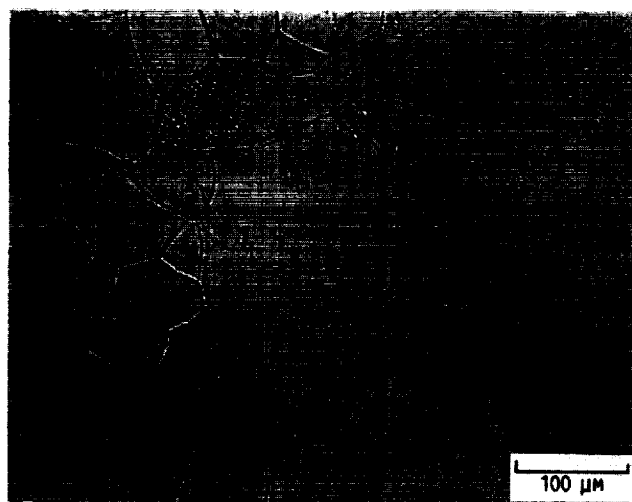


FIGURE 9. - PHOTOMICROGRAPH OF A REGION IN Fe-41.7Al WHERE THE ORIGINAL HIGH ANGLE GRAIN BOUNDARY STRUCTURE IS BEING REPLACED BY A MOSAIC OF LOW ANGLE GRAIN BOUNDARIES. SPECIMEN TESTED AT $1400\text{ K} - \dot{\epsilon} \ 2.4 \times 10^{-4} \text{ s}^{-1}$ TO 30 PERCENT STRAIN AND ETCHED WITH 25 ACETIC ACID, 25 HCl; 50 H₂O AND 1 HF (PARTS BY VOLUME), REF. 17.

ORIGINAL PAGE IS
OF POOR QUALITY

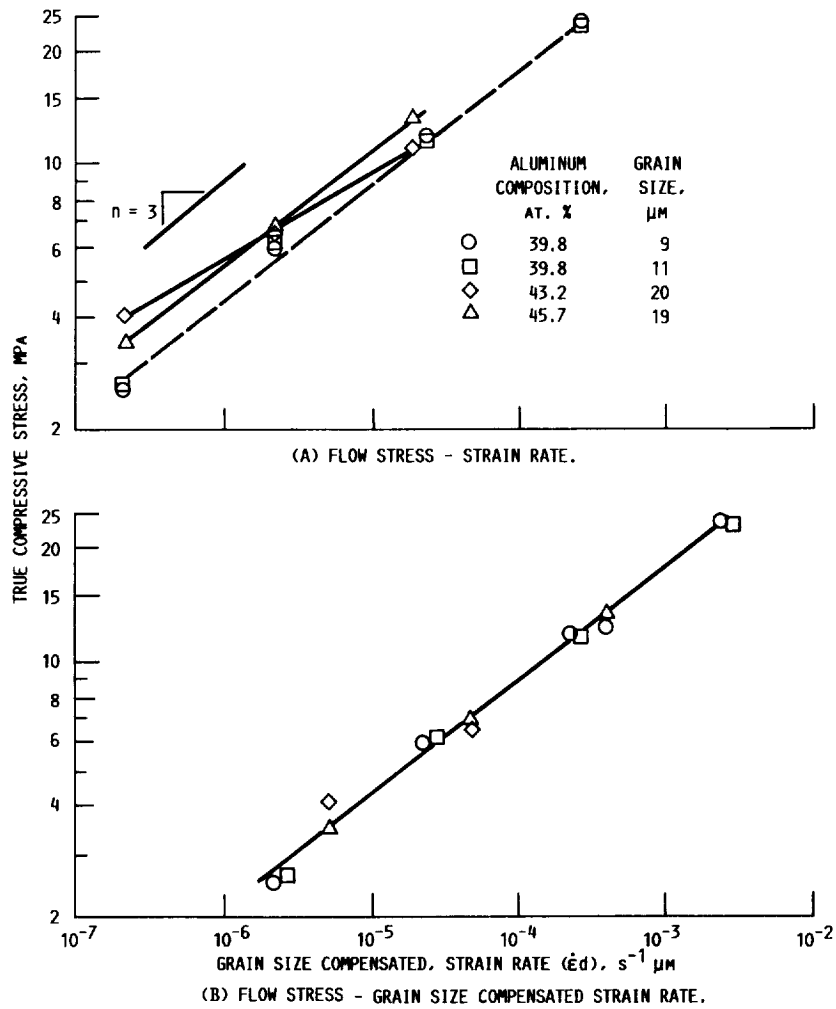


FIGURE 10. - FLOW STRESS - STRAIN RATE AND FLOW STRESS - GRAIN SIZE COMPENSATED STRAIN RATE BEHAVIOR OF B2 IRON ALUMINIDES AT 1300 K IN THE REGIME WHERE GRAIN SIZE WEAKENING OCCURS.

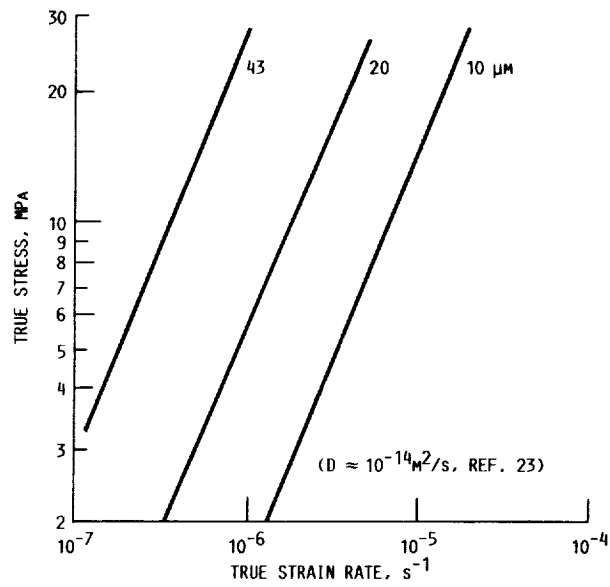


FIGURE 11. - PREDICTED NABARRO-HERRING CREEP RATES IN THE B2 ALUMINIDE FeAl AT 1300 K AS A FUNCTION OF GRAIN SIZE.

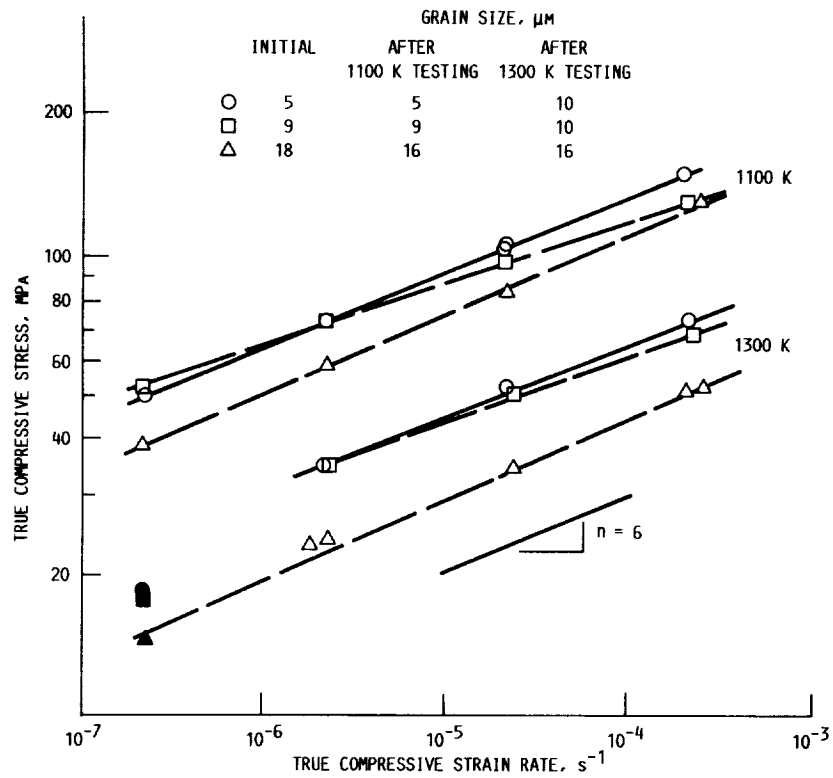


FIGURE 12. - TRUE COMPRESSIVE FLOW STRENGTH - STRAIN RATE BEHAVIOR AT 1100 AND 1300 K FOR Ni-48.25Al AS A FUNCTION OF GRAIN SIZE.

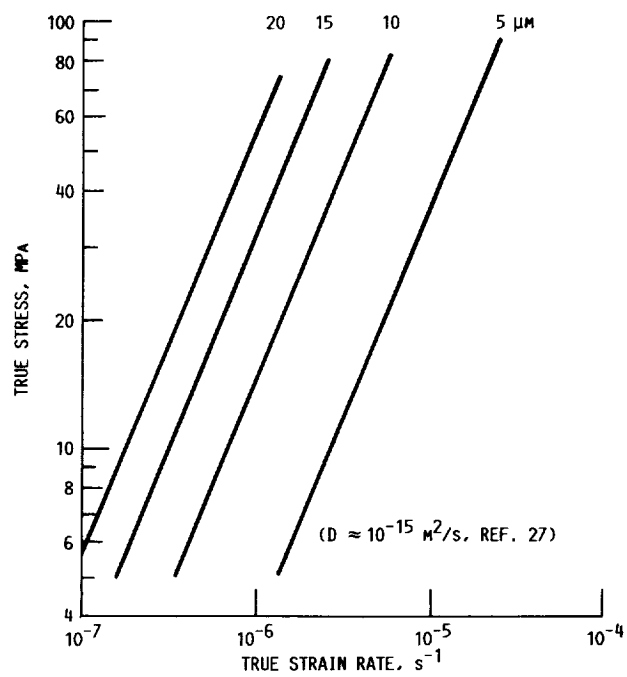


FIGURE 13. - THEORETICAL NABARRO-HERRING DIFFUSIONAL CREEP RATES AT 1300 K IN THE B2 ALUMINIDE NiAl AS A FUNCTION OF STRESS AND GRAIN SIZE.

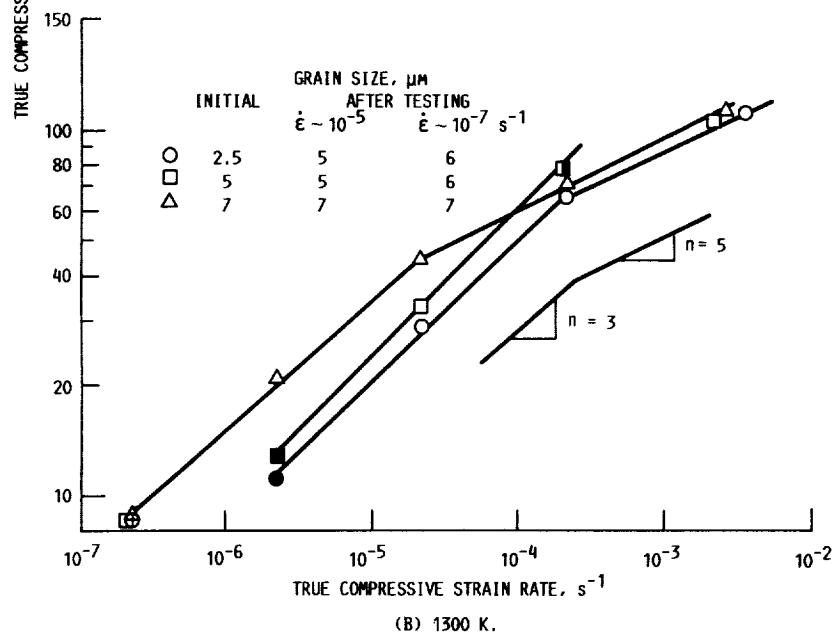
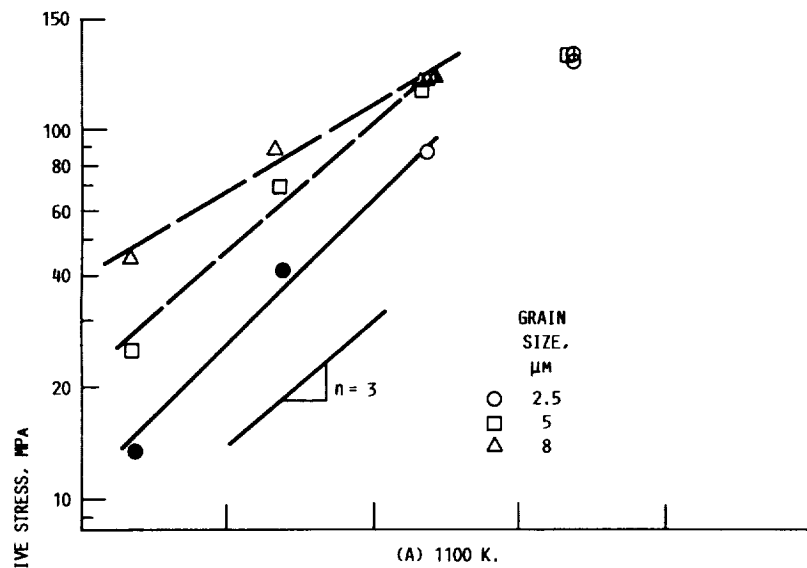


FIGURE 14. - FLOW STRESS - STRAIN RATE BEHAVIOR FOR CoNiAl AS A FUNCTION OF GRAIN SIZE.

Report Documentation Page

1. Report No. NASA TM-101382		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Effect of Grain Size on the High Temperature Properties of B2 Aluminides				5. Report Date	
				6. Performing Organization Code	
7. Author(s) J. Daniel Whittenberger				8. Performing Organization Report No. E-3557	
				10. Work Unit No. 505-63-01	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546-0001				14. Sponsoring Agency Code	
15. Supplementary Notes Presented at the 1987 Northeast Regional Meeting "High Temperature Structural Composites: Synthesis, Characterization and Properties" cosponsored by the Metallurgical Society of AIME and The Materials Research Society, Hoboken, New Jersey, May 27-29, 1987					
16. Abstract Measurements of the slow plastic flow behavior of cobalt, iron and nickel B2 crystal structure aluminides have been conducted on materials fabricated by powder metallurgical techniques. Due to this processing, the aluminides invariably had small equiaxed grains, ranging in size from about 3 to 60 μm in diameter. Grain size was dependent on the extrusion temperature utilized for powder consolidation, and it proved to be remarkably stable at elevated temperature. Mechanical properties of all three aluminides were determined via constant velocity compression testing in air between 1000 and 1400 K at strain rates ranging from $\sim 10^{-3}$ to 10^{-7} s^{-1} . At high temperatures and slow deformations rates decreasing the initial grain size was found to lower the flow stress; however the opposite behavior was generally noted for faster straining or lower temperature conditions, where decreasing the grain size increased the strength. FeAl provides the clearest example of grain size strengthening where Hall-Petch behavior appears to be responsible for the effect. In NiAl increases in the flow stresses are only found when the grain size was $\leq 15 \mu\text{m}$; possibly due to a competition between Hall-Petch and subgrain mechanisms. Decreasing the grain size of CoAl appears to be deleterious and just weakens the material. The transition between grain size strengthening and weakening is, most likely, the result of grain boundary sliding and/or diffusional creep which can become important in small grain sized materials under high temperature, slow strain rate conditions.					
17. Key Words (Suggested by Author(s)) Aluminides; NiAl; CoAl; FeAl; Intermetallics			18. Distribution Statement Unclassified--Unlimited Subject Category 26		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No of pages 26	
				22. Price* A03	

National Aeronautics and
Space Administration

Lewis Research Center
Cleveland, Ohio 44135

Official Business
Penalty for Private Use \$300

FOURTH CLASS MAIL

ADDRESS CORRECTION REQUESTED



Postage and Fees Paid
National Aeronautics and
Space Administration
NASA 451

NASA
